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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART XI ZINC AND ITS ALLOYS AND THEIR EXAMINATION

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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART XI ZINC AND ITS ALLOYS AND THEIR EXAMINATION

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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART XI ZINC AND ITS ALLOYS AND THEIR EXAMINATION

0. FOREWORD

0.1 This Indian Standard (Part XI) was adopted by the Indian Standards Institution on 24 December 1976, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of the microscope. Because of the diversity in available equipment, the wide variety of problems encountered, and the personal element, this standard gives for the guidance of the metallographer only those practices which experience has shown are generally satisfactory. This part covers the polishing, etching and examination of zinc and its alloys.

0.3 This standard is being issued in parts. This part covers polishing, etching and examination of zinc and its alloys. The other parts of this code are as follows:

Part I	General features
Part II	Electrolytic polishing
Part III	Aluminium and its alloys and their examination
Part IV	Copper and its alloys and their examination
Part V	Iron and steel and their examination
Part VI	Lead and its alloys and their examination
Part VII	Magnesium and its alloys and their examination
Part VIII	Nickel and its alloys and their examination
Part IX	Gold, silver, platinum, palladium and their alloys and their examination
Part X	Tin and its alloys and their examination

0.4 In the preparation of this standard assistance has been derived from:

ASTM Designation: E3-62 Standard methods of preparation of metallographic specimens. American Society for Testing and Materials.

Metals handbook. 1973. American Society of Metals.

1. SCOPE

1.1 This standard (Part XI) covers the polishing, etching and examination of zinc and its alloys.

2. PREPARATION OF SPECIMENS

2.0 Recommended methods of selection, size, cutting, cleaning and mounting of specimens are given in IS : 7739 (Part I)-1975*. Recommended methods of electrolytic polishing are given in IS : 7739 (Part II)-1975†.

2.1 There are two facts which should be kept in mind when preparing zinc specimens, particularly the purer grades of zinc namely, (a) mechanical twins are readily formed, especially in coarse-grained zinc. These may be formed during the polishing operation, and care should be taken to remove a sufficient depth of metal in subsequent operations to cut beneath the distorted layer; and (b) the recrystallization temperature of commercial zinc may range from 100°C to as low as room temperature for the purest zinc. Polishing or grinding operations should be carried out under a steady flow of coolant so as to avoid undue heating of the specimen which may enhance further grain growth.

2.2 After mounting, the specimen is ground on a belt sanding machine or a rotary type disc grinding machine using successively finer grit material, for example, 320, 400 and 600 successively. For disc grinding, the maximum speed should not exceed 250 rev/min to avoid overheating. Use of silicon carbide belt or disc is recommended to reduce susceptibility to loading. At least 1·5 mm of the surface of the section should be removed in these operations so that the distortion produced at the sheared or sawed edges of the specimen may be removed. It is advisable to use a continuous water spray or alternatively the specimen may be occasionally dipped in water to avoid heating. (For details of grit numbers of abrasive papers, *see* IS : 715-1976‡. A comparative chart for grit numbers of abrasive grains is also given in Appendix A.)

2.3 Wet polishing (with mineral lubricants, for example, kerosene, paraffin, etc) is carried out on four cloth-covered wheels, rotating at 200 to 400 rev/min. The first two are covered with white duck, the last two with broadcloth or billiard cloth. Abrasives suitable for aluminium and magnesium may be used. Care is also needed in wet polishing to remove the distortion produced by the preceding operation. The amount of metal removed during wet polishing is slight and it is more feasible to

*Code of practice for preparation of metallographic specimens: Part I General features.
†Code of practice for preparation of metallographic specimens: Part II Electrolytic polishing.
‡Specification for coated abrasives (*third revision*).

remove the bulk of the distorted metal by etching with the Palmerton reagent. The following schedule is recommended:

Etch 3½ minutes

Polish on No. 1 wheel

Etch 1½ minutes

Polish on No. 2 wheel

Etch 30 seconds

Polish on No. 3 wheel

Etch 10 seconds

Polish on No. 4 wheel

Etch 3 seconds

2.4 The etching schedule may be varied somewhat depending upon the specimen and the purpose of the examination. For examination at low magnification polishing may generally be stopped at the third wheel. When the oil immersion objective is to be used, the etching time after the fourth wheel should generally be reduced to 1 second; (the time varies from alloy to alloy) particularly if there are constituents present which are unattacked by the etching reagent. Otherwise, the difference in elevation between etched and unetched constituents shall make it impossible to bring both into focus at one time. When polishing specimens containing constituents of widely varying hardness and etching characteristics, such as galvanized iron or plated zinc, alternate polishing and etching is not advisable as the etched structure is not completely removed during the subsequent polishing due to the nap of the cloth.

2.5 Care should be taken to avoid carrying coarse abrasive to a finer wheel. The best means of avoiding this is to wash both the hands and the specimen with soap and water between polishing operations on the wheels. Dragging out of inclusions or cavities may be minimized by occasionally rotating the specimen 180° or even by continuously rotating the specimen.

2.6 An improved method of preparation of specimens of galvanized iron or steel, involves the use of levigated alumina in water with a few drops of triethanolamine added to keep the *p*H value between 7 and 7·6.

3. ETCHING REAGENTS

3.1 In Table 1 are given the etching reagents commonly recommended for zinc and its alloys.

TABLE 1 ETCHING REAGENTS FOR ZINC AND ITS ALLOY

(*Clause* 3.1)

Sl No.	Etching Reagent	Composition*		Remarks	Use
(1)	(2)	(3)		(4)	(5)
i)	Palmerton reagent	CrO_3 (99·95 percent) Na_2SO_4 (c.p.) H_2O	200 g 15 g 1 000 g	Immersion with gentle agitation follow with rinse in solution of: CrO_3 200 g H_2O 1 000 g	General (reduce Na_2SO_4 to 7·5 g when using solution to develop grain structure in alloys containing copper)
ii)	Diluted Palmerton reagent	CrO_3 (99·95 percent) Na_2SO_4 (c.p.) H_2O	50 g 4 g 1 000 ml	Immersion for 2 to 3 s. Follow with rinse in solution of: CrO_3 200 g H_2O 1 000 ml	Structure of die castings, also contrast between the same and plated coating
iii)	Hydrochloric acid	HCl			Macrostructure of pure zinc
iv)	Dilute (0·5 percent) nitric acid (Nital)	HNO_3 alcohol (95 percent) or HNO_3 amyl alcohol	1 drop 10 ml 3 drops 50 ml	Etched by immersion (the reagent should be used within 1 hour after mixing) and subsequent rinsing in absolute ethyl alcohol	For showing the iron-zinc alloy layer in galvanized iron or steel

*The use of concentrated reagents is intended, unless otherwise specified.

3.2 The Palmerton reagent is the most widely used etching solution for zinc alloys; it is followed by the rinse in the chromic acid solution to avoid staining.

4. EXAMINATION AND IDENTIFICATION OF CONSTITUENTS

4.1 After polishing is completed, the finger dipped in alcohol is rubbed lightly over the surface of the specimen to remove any slight film of abrasive. The specimen is then rinsed successively in alcohol, ether, alcohol, and water, the excess water shaken off, and the specimen immersed in the etching solution. After etching, the specimen is immediately and thoroughly rinsed successively in water, alcohol and ether, and dried in a blast of warm air. Nothing should touch etched surface, which is very susceptible to scratching. The etched specimen should be preserved in a desiccator to avoid tarnishing.

4.2 For determination of grain size, the polished specimen should be examined under illumination by properly compensated polarized light, as under these conditions individual grains stand out and grain counts may be accurately carried out. Grain boundaries are poorly defined in white light.

4.3 Specimens to be examined for corrosion should be polished through the fourth wheel, and examined in white light in an unetched condition.

APPENDIX A

(*Clause* 2.2)

COMPARATIVE CHART OF GRIT NUMBERS OF ABRASIVE PAPERS

A-1. For designation of grain sizes of coated abrasives only grit numbers are recognized in IS : 715-1976*. Although grit numbers are now being universally adopted, coated abrasive papers are also available in a graded symbol sequence of 12/0 to 4½ covering the grit number range of approximately 600 to 12. There is also a special emery polishing paper for very fine polishing, such as used in preparation of metallurgical specimens, which is graded in a symbol sequence of 4/0 to 3, covering the grit number

*Specification for coated abrasives (*third revision*).

range of approximately 600 to 180. A comparative chart showing the relation between grit numbers and the customary grading symbols are given below for the guidance of users:

Grit No.	*Symbol for Silicon Carbide Paper*	*Symbol for Emery Polishing Paper*
600	12/0	4/0
—	—	3/0
500	11/0	2/0
460	10/0	0
360	—	—
320	9/0	½
280	8/0	—
240	7/0	1G
220	6/0	2
180	5/0	3
150	4/0	—
120	3/0	—
100	2/0	—
80	0	—
60	½	—
50	1	—
40	1½	—
36	2	—
30	2½	—
24	3	—

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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